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Title:

Corrosion Testing Proposal -1: Assessment of the Corrosion of Stainless Steel Subjected to an Aqueous Solution of Hydrochloric Acid

Jimenez, Stephen Rudy Burleigh, T. David Author(s):

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# **Corrosion Testing Proposal-1:**

Assessment of the Corrosion of Stainless Steel Subjected to an Aqueous Solution of Hydrochloric Acid



Prepared for: U.S. Department of Energy/National Nuclear Security Administration

(NNSA),

Los Alamos National Laboratory (LANL)

David Grow and Paul Smith

Operational Readiness & Execution (ORI-2)

Prepared by: Stephen Rudy Jimenez, Lead R&D Engineer

Operational Readiness & Execution (ORI-2)

T. David Burleigh, PhD, PE

New Mexico Institute of Mining and Technology

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# **Acronyms and Abbreviations**

| Acronym | Definition  |  |  |  |
|---------|---|--|--|--|
| Cu      | Copper  |  |  |  |
| CW      | Cold-worked   |  |  |  |
| CWG     | Corrosion Working Group                                       |  |  |  |
| HCL     | Hydrochloric Acid   |  |  |  |
| HT      | Heat treatment  |  |  |  |
| ID      | Sample Identification Number                                  |  |  |  |
| LANL    | Los Alamos National Laboratory                                |  |  |  |
| NNSA    | National Nuclear Security Administration                      |  |  |  |
| ORI-2   | Operational Readiness & Execution                             |  |  |  |
| SAVY    | Stone, Anderson, Veirs, Yarbro (first initial of the four co- |  |  |  |
|         | developers of specialized container)                          |  |  |  |
| TBD     | To Be Determined  |  |  |  |



## 1 The Necessity for Corrosion Testing of Stainless Steel

This corrosion-test proposal outlines the methods to promote rapid pitting corrosion on various pitting resistant stainless steels under controlled conditions. This study can be performed in-whole or inpart as the Corrosion Working Group (CWG) leadership of the Los Alamos National Laboratory (LANL) deems pertinent. This proposal is in response to CWG's need to understand the mechanisms and the possible detrimental failure of the 316L SAVY (an acronym that represents the first initial of the four codevelopers, Stone, Anderson, Veirs, Yarbro) 4000 container design currently in service. This detrimental failure evolved from a question that the CWG arrived to by consensus as "What is the critical pit depth that would comprise the design wall-strength of the SAVY-4000?" Numerous measuring techniques have been used to measure pit depths i.e. thermal imaging, ultrasonic, eddy currents, and laser confocal microscope [1, 2, and 3]. However, because pits can grow and expand 3-dimensionally and multi-directional, there is no current technique that can accurately measure the depth of a pit that has grown horizontally and then vertically to perforation like in undercutting and horizontal grain attack, see figure 1 [4].

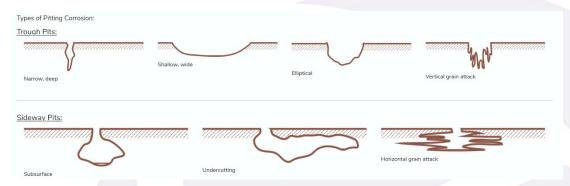


Figure 1. Schematic of various pits.

### 1.1 Definition of Critical Pit Depth

The optimal definition of critical pit depth should be redefined as "the critical pit depth equals total thickness of the wall", and in this case is approximately 0.03". This would be failure and a danger to personnel and the environment. Previous corrosion tests have been performed on 316L at LANL and the New Mexico Institute of Mining and Technology (NMT). These studies have shown that when subjected to hydrochloric acid (HCl) and humidity, 316L samples were vigorously attacked by pitting corrosion. Molar concentrations of HCl for these studies were 3M and 6M.

#### 1.2 The Purpose of Assessment

The purpose of this study is to determine the total time needed for HCL to breakdown the passivation layer of the stainless steel and corrode through a predetermined thickness. These results will be presented as a range of thicknesses of samples versus Molar concentration of HCL. These thicknesses will start at the thinnest, 0.005", and increment up to 0.06" for a total of 6 different thicknesses per stainless steel. The thickness of the SAVY wall of 0.03" will be included in the aforementioned range. The results will be compared to samples that best represent the thickness of the SAVY wall. There will be three different Molar concentrations of HCl used for this study: 1) 1M, 2) 3M, and 3) 6M. Along with 316L, there will be an additional two types of corrosion-resistance stainless steels suggested by the



corrosion handbook. These alloys are Inconel 625 and Hastelloy C22 [5]. These corrosion resistant stainless steels along with 316L will be purchased from Alloy International, Inc.

#### 1.3 Test Method

The test method for this study has been used and suggested by Dr. T. David Burleigh P.E. of NMT (see Figure 2). The details of this particular method are as follows:

- 1. All samples will be cut to a 3" x 3" squares.
- 2. A 25mm diameter O-ring glass cylinder containing the HCl solution will be clamped to the stainless steel sheet.
- 3. In order to detect when the stainless steel has been perforated by pitting, the stainless steel sheet will be separated from a copper (Cu) sheet by a thin piece of paper. When the HCl perforates the stainless steel, the electrolyte will soak the paper, completing an electrical circuit, and lighting a light or stopping a timer.



Figure 2. Proposed accelerated test configuration.

The paper sheet would be electrically insulating, until the HCl exudes from the perforated pit and soaks the paper sheet. At this point there would be electrical contact between the stainless steel above and the Cu electrode below. With a battery and a light, buzzer or timer, it would signal when there is electrical conduction and thus perforation. The time for perforation will be recorded and will be correlated to the sample identification (ID) number, see Table 1. The ID number will identify the type of stainless steel, sheet thickness in inches and HCl molar concentration.

#### 1.4 Test Results

The results will be graphed in perforation thickness vs. time for each HCl concentration and stainless steel type. Figure 3 shows the expected format for this data. We plan to be able to estimate the depth of pitting versus time for each concentration of HCl. For statistical soundness, six (6) samples will be used for each stainless type, thickness and HCl concentration, totaling 270 individual samples (see Table 2). The heat treatment (HT) of these set of samples will be "to be determined" (TBD) from the vendor and this information will be added to the IDs. A second set of 270 samples mirroring the above proposed test matrix will be cold rolled (cold worked, CW) by 50% by an outside vendor and re-tested by NMT. A small set of one or both of the "as received" and CW groups will be sensitized (~600 C for 10 hr.) and exposed to the same regimen of HCL exposure as the larger groups of samples. This HT set will be used to serve this study as an extreme conditions data set.



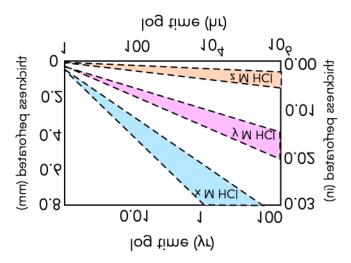


Figure 3. Planned format of expected data for each stainless steel.

The results from this study will be used for comparison when LANL's future studies can predict/measure accurately amounts of HCl, and compared to any section of the current SAVY container that was cold worked during manufacturing. This will assist LANL corrosion working group to determine a design life extension, if any, on the SAVY 4000. The addition of alloys Inconel 625 and Hastelloy C22 will offer the LANL container design and corrosion teams creditable options of stainless steel for future SAVY container design.

All the work for this study will be performed at NMT under the supervision and guidance of Dr. David Burleigh, P.E. Therefore, all materials and necessary supplies for the study will be shipped to NMT from the vendor to Dr. Burleigh.



# 2 Sample Test Matrices

Table 1: As Received Sample ID Test Matrix

| SS Type              | Approximate<br>Thickness<br>(in) | Sample IDs<br>(1M) | Sample IDs<br>(3M) | Sample IDs<br>(6M) | Total No. of<br>Samples per<br>Thickness |
|----------------------|----------------------------------|--------------------|--------------------|--------------------|--|
| 316L                 | 0.005                            | 316L-005-<br>1M    | 316L-005-<br>3M    | 316L-005-<br>6M    | 18                                       |
|                      | 0.01                             | 316L-01-1M         | 316L-01-3M         | 316L-01-6M         | 18                                       |
|                      | 0.02                             | 316L-02-1M         | 316L-02-3M         | 316L-02-6M         | 18                                       |
|                      | 0.03                             | 316L-03-1M         | 316L-03-3M         | 316L-03-6M         | 18                                       |
|                      | 0.06                             | 316L-06-1M         | 316L-06-3M         | 316L-06-6M         | 18                                       |
|                      |                                  |                    |                    |                    |  |
| 625                  | 0.005                            | 625-005-1M         | 625-005-3M         | 625-005-6M         | 18                                       |
|                      | 0.01                             | 625-01-1M          | 625-01-3M          | 625-01-6M          | 18                                       |
|                      | 0.02                             | 625-02-1M          | 625-02-3M          | 625-02-6M          | 18                                       |
|                      | 0.031                            | 625-031-1M         | 625-031-3M         | 625-031-6M         | 18                                       |
|                      | 0.063                            | 625-063-1M         | 625-063-3M         | 625-063-6M         | 18                                       |
|                      |                                  |                    |                    |                    |  |
| C-22                 | 0.005                            | C22-005-1M         | C22-005-3M         | C22-005-6M         | 18                                       |
|                      | 0.01                             | C22-01-1M          | C22-01-3M          | C22-01-6M          | 18                                       |
|                      | 0.02                             | C22-02-1M          | C22-02-3M          | C22-02-6M          | 18                                       |
|                      | 0.032                            | C22-032-1M         | C22-032-3M         | C22-032-6M         | 18                                       |
|                      | 0.063                            | C22-063-1M         | C22-063-3M         | C22-063-6M         | 18                                       |
| Total of No. samples | C.1.: . 1.1 . :11.1              | 0                  | 0                  | 0                  | 270                                      |

<sup>\*</sup>Note: A CW version of this table will be implemented that would have the –CW suffix designation to the IDs, i.e. 316L-005-1M-CW. A small subset if samples will be identified for HT and will be identified by adding the suffix –HT at the end of the sample IDs, i.e. 316L-005-1M-HT.



Table 2: As Received Sample Test Matrix

| SS Type              | Approximate<br>Thickness<br>(in) | No. of<br>Samples per<br>1M HCL | No. of<br>Samples per<br>3M HCL | No. of<br>Samples per<br>6M HCL | Total No. of<br>Samples per<br>Thickness |
|----------------------|----------------------------------|---------------------------------|---------------------------------|---------------------------------|--|
| 316L                 | 0.005                            | 6                               | 6                               | 6                               | 18                                       |
|                      | 0.01                             | 6                               | 6                               | 6                               | 18                                       |
|                      | 0.02                             | 6                               | 6                               | 6                               | 18                                       |
|                      | 0.03                             | 6                               | 6                               | 6                               | 18                                       |
|                      | 0.06                             | 6                               | 6                               | 6                               | 18                                       |
|                      |                                  |                                 |                                 |                                 |  |
| Inconel 625          | 0.005                            | 6                               | 6                               | 6                               | 18                                       |
|                      | 0.01                             | 6                               | 6                               | 6                               | 18                                       |
|                      | 0.02                             | 6                               | 6                               | 6                               | 18                                       |
|                      | 0.031                            | 6                               | 6                               | 6                               | 18                                       |
|                      | 0.063                            | 6                               | 6                               | 6                               | 18                                       |
|                      |                                  |                                 |                                 |                                 |  |
| Hastelloy C-22       | 0.005                            | 6                               | 6                               | 6                               | 18                                       |
|                      | 0.01                             | 6                               | 6                               | 6                               | 18                                       |
|                      | 0.02                             | 6                               | 6                               | 6                               | 18                                       |
|                      | 0.032                            | 6                               | 6                               | 6                               | 18                                       |
|                      | 0.063                            | 6                               | 6                               | 6                               | 18                                       |
| Total of No. samples | C.1 11                           | 90                              | 90                              | 90                              | 270                                      |

<sup>\*</sup>Note: A CW version of this table will be implemented that would total (270 x 2) 540 samples. The quantity of a small subset if samples will be identified for HT will be TBD. As a result, the total number of samples for this study will be 540+.

## 3 References

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